California Environmental Protection Agency Department of Toxic Substances Control Hazardous Materials Laboratory SOP No.: 914-S Revision No.: 2

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SOP Status: Operational	Approved by:
	original signed by Bart Simmons, HML Chief
original signed by Cindy Dingman, QC Officer	original signed by Jarnail Garcha, Supervisor

Procedural SOP No. 914-S

Preparation of Cold Cathode Fluorescent Lamps for Mercury Testing, including WET and TCLP

1. Scope and Application

This SOP is applicable to the preparation of cold cathode fluorescent lamps (CCFL) for mercury analysis using EPA Method 7470A, 7471A, EPA Method 1311 for TCLP, and HML Method 910-M for WET. CCFLs are commonly used in liquid crystal display (LCD) electronic devices.

2. Safety

- 2.1. Protective nitrile gloves and a face shield should always be worn while crushing the samples.
- 2.2. Crushing of the samples should always be carried out in the hood.
- 2.3. Samples should be wrapped in double heavy duty tear resistant plastic bags before crushing.

3. Materials and Equipment

- 3.1. Heavy duty hydraulic press, 40000 lb RAM force, 4" RAM (Pasadena Hydraulics, Inc.), or equivalent.
- 3.2. Polypropylene tear resistant plastic bags that can withstand 165 g dart test per ASTM D1709-85 (1.5 X 2 ft).
- 3.3. Rubber Mallet or hammer.
- 3.4. Sieves No.18 mesh (1 mm opening) and No. 10 (2 mm opening).

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- 3.5. Glass containers.
- 3.6. Freezer (-12 ⁰ C).
- 3.7. Scissors or Wire cutter.
- 3.8. Mortar and Pestle.

4. Procedure:

- 4.1. For Total Mercury, TCLP and WET Determinations
 - 4.1.1. Cut the end cap wiring attached to the lamp with the scissors or a wire cutter. Record the weight and store separately or save the wiring with the metal fraction of the device, if appropriate, as described in HML SOP 916-S. Store samples at minus 12 °C.
 - 4.1.2. Weigh and record the weight of each lamp (or all lamps for a composite sample, if TCLP and or WET analysis is required) along with the end caps.
 - 4.1.3. Place the lamp with the end caps intact into a double heavy duty polypropylene plastic bag. For longer lamps use extra long bags. Leave the sample containing bag in a freezer for one hour.

Note: Do not remove the end caps or break the sample before freezing.

- 4.1.4. Take the frozen sample (in the plastic bag) out of the freezer and break the lamp initially with a rubber mallet or a hammer into small pieces, then crush the lamp under the hydraulic press (if necessary).
- 4.1.5. Transfer the crushed samples from the plastic bag into a mortar and grind with the pestle until all the materials pass through the 1mm sieve for total Hg analysis, and use the 2mm sieve for WET & TCLP. Weigh and set aside the visible small end cap copper wire pieces.
- 4.1.6 Weigh and transfer the sieved sample into a glass container and store at -12⁰ C.
- 4.1.7. Take an aliquot of 0.2 to 1.0 gram of the above prepared sample for total Hg analysis by EPA Method 7471A (or use the entire sample if necessary, to meet the detection limit criteria for this analysis). Test sub-samples in triplicate.

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- 4.1.8. If enough sample material is available, take an aliquot of the sample from step 4.1.6 of the above procedure for WET and TCLP analysis.
- 4.1.9. Five to ten grams of sample may be used for WET and /or TCLP, based on sample availability. Add a proportionate amount of extracting fluids to the sample and perform WET and/or TCLP extractions as outlined in HML Method 910-S and EPA Method 1311, respectively, and determine Hg concentrations by EPA Method 7470A.

Important Note: For WET and TCLP, use extraction vessels that can accommodate the sample and the extraction fluid with as little head space as possible to avoid any loss of Hg due to dissipation or evaporation. Digest the extracts right after the extraction. Mercury may dissipate or evaporate in the head space if the extracts are stored for an extended period of time.

5. References

- 5.1. California Code of Regulations, Title 22, Vol. 29, Article 11, Sections 66699, 66700.
- 5.2. Toxicity Characteristic Leaching Procedure, Federal Register, Method 1311, SW-846.
- 5.3. Test Methods for Evaluating Wastes: Physical/Chemical Methods, US Environmental Protection Agency, Office of Solid Waste Washington, DC, SW846, Vol. 1A, 3rd Edition, Update III.

6. Acknowledgement

This procedure was developed by the Inorganic Section of the Hazardous Materials Laboratory, Department of Toxic Substances. For more information please contact Jarnail Garcha at (510) 540-3468.